

10/527 441

PTO 08-0570

CC=RU DATE=19950709 KIND=C1  
PN= 2039125

COMPOSITION POWDER FOR PLASMA SPUTTERING OF COATINGS  
[COMPOZITSIONNYY POROSHOK DLYA PLAZMENNOGO NAPYLENIYA POKRYTIY]

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UNITED STATES PATENT AND TRADEMARK OFFICE  
Washington, D.C. October 2007

Translated by: FLS, Inc.

PUBLICATION COUNTRY	(10):	RU
DOCUMENT NUMBER	(11):	2039125
DOCUMENT KIND	(12):	C1
PUBLICATION DATE	(46):	19950709
APPLICATION NUMBER	(21):	5009968/02
APPLICATION DATE	(22):	19910815
INTERNATIONAL CLASSIFICATION	(51):	C23C 4/08; B22F 1/02
PRIORITY COUNTRY	(33):	NA
PRIORITY NUMBER	(31):	NA
PRIORITY DATE	(32):	NA
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TITLE	(54):	COMPOSITION POWDER FOR PLASMA SPUTTERING OF COATINGS
FOREIGN TITLE	[54A]:	COMPOZITSIONNYY POROSHOK DLYA PLAZMENNOGO NAPYLENIYA POKRYTIY

The invention concerns powder metallurgy, in particular materials for plasma sputtering of protective and wear-resistant coatings.

The problem of hardening the surface of different articles has great significance, since the application of wear-resistant, heat-resistant, and other protective coatings makes it possible to produce construction materials with high usage properties, which sharply increases the service life of the articles. Most often composition powders are used as a material to be sputtered onto the surface of a material by the plasma method.

A composition powder, which includes aluminum, chromium, molybdenum, coated with a layer of aluminum, is known. The thickness of the nickel coating is 5-10  $\mu\text{m}$ . The results of investigations of plasma coatings, produced from the indicated powder, show that the porosity of a coating 0.2 mm thick reaches 10% and the strength of the adhesion of a coating 0.2 mm thick with a St3 steel base is 25-29 MPa.

The closest technical solution is a composition powder for plasma sputtering which has the following composition: nickel 10-12 % by weight, the rest titanium, particle diameter 45-100  $\mu\text{m}$ .

The strength of adhesion of the powder, obtained by plasma sputtering of powdered Ti-Ni with steel is 30-35 MPa in the case of a thickness of 0.4 mm. The hardness of the coating is on the order of 40 HRC units, and the microhardness of the  $\text{Ni}_3\text{Ti}$  phase is on the order of 6900 MPa. A Ti-Ni-based coating is used basically as a wear-resistant coating.

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\*Numbers in the margin indicate pagination in the foreign text.

The fact that the coating obtained from plasma sputtering has a porosity of 2-10% and has a complex phase composition is a disadvantage of the composition powder. Titanium-nickel alloys, titanium oxide ( $\text{TiO}_2$ ), and complex oxides ( $\text{NiTiO}_3$ ) are present in the coating. A coating of powdered Ti-Ni is used basically as a wear-resistant coating, operating in conditions of wear without impact loads. In addition, the coating has low heat resistance and it oxidizes intensively in the case of heating above  $350^\circ\text{C}$ .

The goal of the invention is to increase the quality of composition powders, providing an increase in the wear resistance, microhardness, and heat resistance of the coating.

A composition powder for plasma sputtering of coatings, having the following ratio of components, by weight: nickel 20-70, aluminum 5-20, the rest titanium, is proposed for achieving the object proposed.

The powder having a particle diameter of 45-120  $\mu\text{m}$  is titanium, first plated with nickel by means of chemical nickel plating, and then with aluminum by the method of thermal decomposition of an organic aluminum compound.

The coating obtained by plasma sputtering of the composition powder Ti-Ni-Al, has a strength of adhesion with a steel base of 55-60 MPa in the case of a coating of 0.4 mm, and the porosity of the coating does not exceed 2-3%. A structural and phase analysis of the coating revealed the presence of an intermetallide phase  $\text{Ni}_3\text{TiAl}$ . The microhardness of the coating amounts to 30000-35000 MPa. Abrasion-

testing of the coating was conducted according to the "block-disk" system at a pressure of 10 MPa and a sliding rate of 8 m/s. The wear rate is equal to  $(10-13) \times 10^{-5}$  g/mm<sup>2</sup>/h. The rate of wear of the coating after heating to 1000°C and holding for 4 h is  $(15-16) \times 10^{-5}$  g/mm<sup>2</sup>/h. The coating was tested in industrial conditions on mandrels for hot coiling of springs in conditions of heating samples to a temperature of 800-850°C. The wear of the samples is 0.035 mm/h.

The percentage content of components and the order of arranging the layers of plating metals are essential distinguishing features of the composition powder.

A powder for gas-thermal sputtering of coatings based on aluminum plated with nickel, Al-Ni, is known. The coating obtained by heating Al-Ni powder has a heterogeneous phase structure and in addition to intermetallide compounds and solid solutions also contains aluminum oxide Al<sub>2</sub>O<sub>3</sub>, entering the coating from the initial aluminum powders. Coatings produced from plated Ni-Al powders have a low microhardness, in the range of 4000-9000 MPa. The microphase nature of the coating often leads to destruction of its layering, which limits the conditions and range of the operating temperatures.

A method of applying an aluminum coating onto a bundle of thread-like crystals for producing a semi-finished product for making a known composition material and sputtering onto the surface of an aluminum layer blank in order to improve the friction conditions between the surfaces of the blank and the instrument is known. However, on the basis of the available data it was impossible to assume that the

combination of the Ti-Ni-Al plating layers and their content make it possible to obtain a coating having high usage properties.

The known composition of the Ti-Ni layer according to the prototype provides a coating with a microhardness on the order of 7000 MPa, and an Al-Ni combination of 9000 MPa, correspondingly while the Ti-Ni-Al composition power claimed has a microhardness of 35,000 MPa.

This is connected with the high reaction capacity of the pyrolytic aluminum and the order of arrangement of the plating layers. Performing the aluminum coating of the TiNi powder in an inert atmosphere excludes the presence of an  $\text{Al}_2\text{O}_3$  oxide layer on the TiNi-AL interface. In addition, the AOS solution penetrates through the pores of the nickel coating and reduces the oxide films on the nickel and titanium. The absence of oxide films on metals and high reaction capacity of the aluminum ensures an energy reaction of the metals up to the melting temperature of aluminum. According to the DTA data, the interaction begins at a temperature  $< 600^\circ\text{C}$ , in this case the liberated heat of the reaction of aluminum with nickel contributes to the interaction with the formation of  $\text{Ni}_3\text{TiAl}$  intermetallide in the /4 during the plasma sputtering.

Example 1. Chemical nickel coating of the titanium powder is performed according to the known method. 50 g of powdered titanium are mixed in a reaction vessel with an alkaline solution (pH 8-9), containing 75 g of nickel sulfate,  $\text{NiSO}_4$ , 83 g of sodium citrate and 83 g of ammonium chloride. At a temperature of  $78-88^\circ\text{C}$  75 g of the reducing agent sodium hypophosphite  $\text{NaH}_2\text{PO}_2$  are added to the reaction

mixture with mixing by portions. The plated powder is removed after the end of the process, washed with water, and dried. Produced are 64 g of powdered Ti-Ni with a nickel content of 20% by weight.

The aluminum coating of the particles of nickel-plated titanium Ti-Ni is performed by decomposition of diisobutyl aluminum hydrides  $(i-C_4H_9)_2AlH$  (DIBAH). 50 g of Ti-Ni particles preliminarily degreased in a mixture of solvents (acetone + alcohol) and dried at  $T = 80^\circ C$  are put into a pear-shaped flask in the atmosphere. 46 ml of a 75% solution of DIBAH in toluene are added, a UZDM-2T ultrasonic generator is put into the flask, and it is switched to a frequency of 22 kHz. The reaction mixture is mixed in the ultrasonic field for 1 h at a temperature of  $250-280^\circ C$ . After pyrolysis has ended, which is determined by the ending of the liberation of gas in a bubbler at the outlet from the reaction vessel, the powder is cooled in an argon flow and held in a vacuum of 0.3 mm Hg for complete removal of the pyrolysis products. Obtained is 56.8 g of powdered titanium, containing, in % by weight, Ni 25, Al 12, and Ti the rest.

We used the powder obtained having the composition, in % by weight, of Ni 25, Al 12, and the rest Ti, with a particle diameter of 45-120  $\mu m$ , as a composition powder for applying the coating onto a steel surface in the following conditions: arc voltage 40-60 V; current force 300-350 A; power 30 kW, plasma-forming gas Ar + 10%  $N_2$ ; gas consumption 2-3  $m^3/h$ , sputtering distance 100-150 mm. A coating 0.4 mm thick having the following properties was obtained: density

98%, microhardness 32000 MPa; wear-resistance  $12 \times 10^{-5}$  g/mm<sup>2</sup>/h; heat resistance (wear after heating to 1000 °C)  $15 \times 10^{-5}$  g/mm<sup>2</sup>/h.

Powders having a different percentage content of components were produced analogous to example 1; the data are given in the table.

A coating having a microhardness 5 times greater than in the case of a coating produced from a powder according to the prototype may be produced on the basis of the data presented. The coating has a high density, high strength of adhesion with the base, a one-phase phase composition, and uniform distribution of hardness along the cross-section of the coating.

#### Formulation of Invention:

Composition powder for plasma sputtering of coatings, containing titanium particles, plated with a nickel layer, having a particle diameter of the composition powder of 45-120 μm, wherein the titanium particles additionally contain a layer of aluminum, located directly on the nickel layer, having the following ratio of components in the composition powder, in % by weight:

aluminum 5-20

nickel 20-70

titanium the remainder



Example	Powder composition, % by weight			Coating characteristics			Indicators of achieving goals		
	Ti	Ni	Al	Density, %	Thickness, $\mu\text{m}$	Adhesion strength, MPa	Micro-hardness, MPA	Wear resistance, $\text{g}/\text{mm}^2 \text{ h}$	Heat-resistance (wear after heating to $100^\circ\text{C}$ for 4 h $\text{g}/\text{mm}^2 \text{ h}$ )
1	45	50	5	97	0.4	56	31000	$12 \times 10^{-5}$	$16 \times 10^{-5}$
2	40	40	20	98	0.4	60	35000	$10 \times 10^{-5}$	$15 \times 10^{-5}$
3	65	20	15	98	0.4	58	33000	$11 \times 10^{-5}$	$15 \times 10^{-5}$
4	20	70	10	97	0.4	57	32000	$11 \times 10^{-5}$	$15 \times 10^{-5}$
5	38	60	2	93	0.4	40	28000	$16 \times 10^{-5}$	$29 \times 10^{-5}$
6	35	30	35	91	0.4	30	23000	$18 \times 10^{-5}$	$25 \times 10^{-5}$
7	75	10	15	92	0.4	32	22000	$20 \times 10^{-5}$	$28 \times 10^{-5}$
8	13	80	7	93	0.4	30	23000	$16 \times 10^{-5}$	$20 \times 10^{-5}$
9*	50	36	14	91	0.4	25	15000	$18 \times 10^{-5}$	$30 \times 10^{-5}$
According to prototype	According to prototype	According to prototype	According to prototype	90	0.4	5	6900	$5 \times 10^{-5}$	$25 \times 10^{-5}$

\* Ti-Al-Ni composition powder (titanium initially plated with aluminum, then nickel)